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Determination of Mechanical Properties of Porous Silicon with Image Analysis and Finite Element

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Abstract

In order to create equivalent images, a series of SEM micrographs of porous silicon were treated with the image analysis procedure, developed using public domain software "ImageJ". A morphological description was used to reduce the complexity of the microstructure of porous silicon and an image analysis procedure has been established to quantify different geometrical parameters related to the shape, size and orientation distribution. This description allows performing predictive calculation of mechanical properties of porous silicon using finite element analysis. Results are compared with experiment and a good agreement is observed

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***Keywords*:** Porous Silicon; Image Analysis; micrographs; Finite element model; mechanical properties.

1. Introduction

The technique used for fabricating layers of porous silicon (PS) with various morphologies is very simple. Porous silicon is easily produced by electrochemical etching in a hydrofluoric (HF) acid solution.

The layers with different porosity and thickness [1] can be obtained by varying anodization current density, HF concentration, time, doping level of silicon substrate, illumination conditions during etching and temperature. The pore diameter can be controllably varied from a few nanometers up to several hundred nanometers by adjusting current density and surface resistivity.

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Porous silicon has been the subject of many studies since the discovery of the strong and visible photoluminescence (PL) in 1990 by Canham [2] because of the potential applications in optoelectronics. Based on its properties to change optical parameters with changing porosity PS can be used to design antireflection coating of solar cells, waveguides, resonators…The efficient luminescence of PS has only been observed on highly porous layers and unfortunately PS with the high porosity is unstable. The cracking is observed when the capillary stresses are very important during the evaporation of the electrolyte [3]. This is the reason why examining the changes in mechanical properties is so important.
In digital circuits, insulating dielectrics separate the conducting parts (wire interconnects and transistors) from one another. The dielectric constant of the porous material can be reduced by increasing the porosity of the film [4]. It is known that, as soon as fabrication starts, PS begins to entrap species from the ambient air. However, oxidation has been used to stabilize luminescent properties of porous silicon and previously PS oxidation was used to create dielectric isolation of integrated circuit [5]. Nevertheless, mechanical properties of porous silicon especially of high porosity are important parameters [4].
The aim of the present work is to report the results of an image analysis procedure, to quantify the different geometrical parameters [6] related to the shape, size and orientation distribution of the pores. Equivalent image obtained by treating SEM micrographs, scanning electron microscopy of porous layers are used to carry out finite element calculation, in order to estimate the mechanical properties of porous silicon.

2. Experimental

The sample PS248 is n-type (100) 4-6 Ωcm resistivity of 59% porosity and 5.2 µm thick [7]. He is aged 12 years and stored at ambient air. Porous silicon is a sponge-like crystalline Si network containing nanometer-sized Si crystallites. Porosity p was the primary variable of the systematic study and was determined by the gravimetric method using the following equation, $p = (m_1-m_2)/ (m_1-m_3)$, where m_1 and m_2 denote respectively the weight before and after formation of the porous layer. m_3 is the weight after removal of the porous layer in a 5% KOH solution. The thickness t of the layer is then determined by, $t = (m_1-m_3)/ S\rho$, where ρ is the density of the bulk silicon, and S the wafer area exposed to HF during anodization. The shape of the pores depends on the doping level of the original substrate. SEM micrographs of the top surface were then performed with a Hitachi 2460N with variable pressure of 1 to 270 Pascals with a gun thermo-ionic emission of a tungsten cathode. It is equipped with a micro-analysis of Kevex Sigma X with a lithium-doped silicon diode of 10 mm square and spectral resolution of 146 eV. A plate-cooled effect Pelletier to -10 ° C is also available.
Figure 1 show the appearance of the porous layer, size, shape and pore distribution can be analyzed showing an even distribution and uniform pore more or less spherical with an average diameter ranging from 4.57 µm and 2 microns.
The cross section of the sample shows the spherical shape of pores along a thickness estimated at approximately 5 microns. This SEM image reveals that it is a macroporous layer. The silicon matrix (gray contrast) and pores (dark contrast) can be clearly distinguished as the main structural element (fig1a).
The testing device is a Fischerscope H100 XYp micro-indenter (maximum load of 1 N, load resolution of 0.02 mN, depth resolution of 2 nm) [4]. Indentation consists in continuously applying a load to a specimen with a sharp Vickers pyramid indenter of face angle 136° and continuously monitoring the penetration depth in the sample. The force is generated electromagnetically and is incrementally increased stepwise up to the peak load of 10, 300

and 1000 mN. The calibration of the instrument was done by using ISO-14577 procedure and with the help of a reference block.

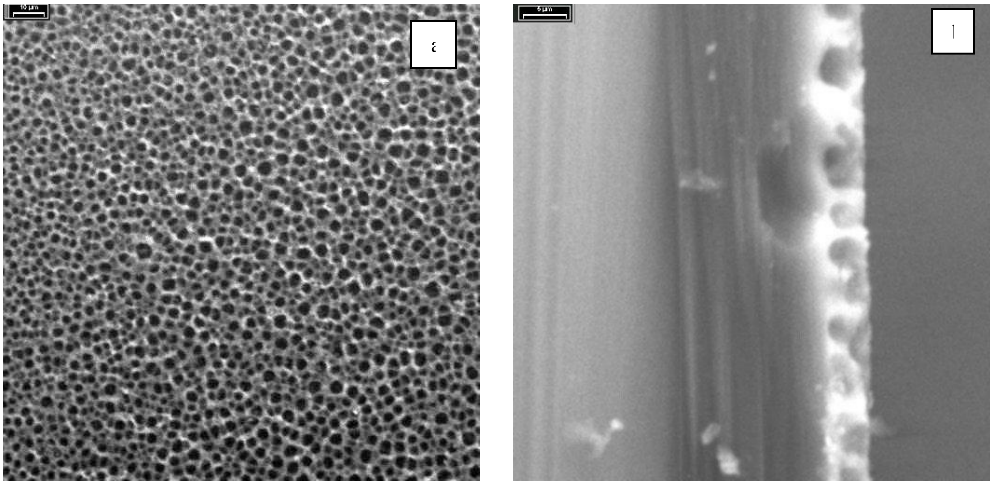

Fig.1 SEM micrograph of the top surface of the 5 µm-thick PS film (a) and cross section (b). The porosity was initially about 59%.

3. Image analysis

A simple way to represent the morphology would be to replace the elements of the microstructure by simplified geometric shapes. Each phase may be replaced by a set of squares or circles with dimensions equal to the average found on the lengths of the structural elements related to this phase. It is possible to represent an image by a set of equivalent ellipse (Figure2).

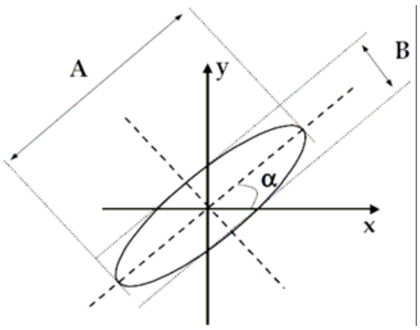

Fig 2. Geometric parameters of an ellipse.

The three parameters related to shape, orientation and size of the ellipses for a statistical analysis of the distribution of porosity equivalent of an image are defined as follows: (i) shape factor: $F_{sh}=A/B$; it corresponds to the ratio between the major axis and minor axis and is equal to unity for a circle. (ii) slope factor: $F_{sl}=\alpha$; it corresponds to the orientation angle of the major axis relative to the parallel direction to the substrate. (iii) size factor:
$F_{SZ} = A * B$; it is the area of the ellipse to factor 3/4 π . F_{SZ} contains information on particle size.

Figure 3 shows a description of obtaining a binary image on the example of a macroporous silicon sample. The image analysis procedure was developed using a public domain software "ImageJ" [8].
We get an image of 512 × 512 pixels, 8 bit, 256 K. This corresponds to ≈341.33 pixels for 100µm. Fig3b represents the corresponding grey level histogram used for the separation of phases with the presence of three colors: black pores represented in dark black, the white represents the effect of the rough parts of the surface illuminated enough to appear as a third phase represented in light gray. It is also possible to represent the effect of roughness from the distribution of gray levels on the surface of the image as can be seen clearly in Figure 4. A manual adjustment is necessary. Pores must be entirely in black .The image must be filtered; a simplification of form is obtained by the command "Despeckle".

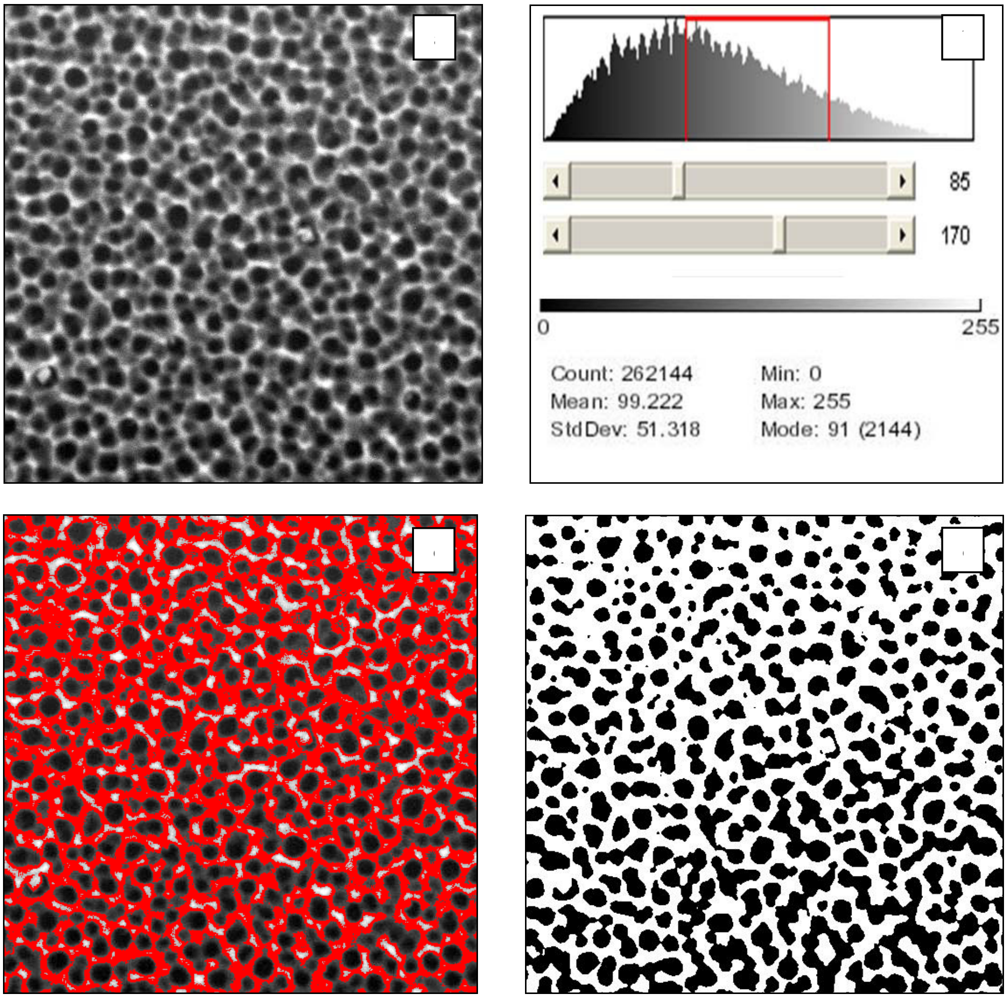

Fig 3. Description of the image analysis procedure: (a) SEM micrograph corresponding to the initial image of porous silicon, (b) corresponding gray level histogram used for the separation of phases, (c) analyzed image, (d) binary Image showing the separated porosity.

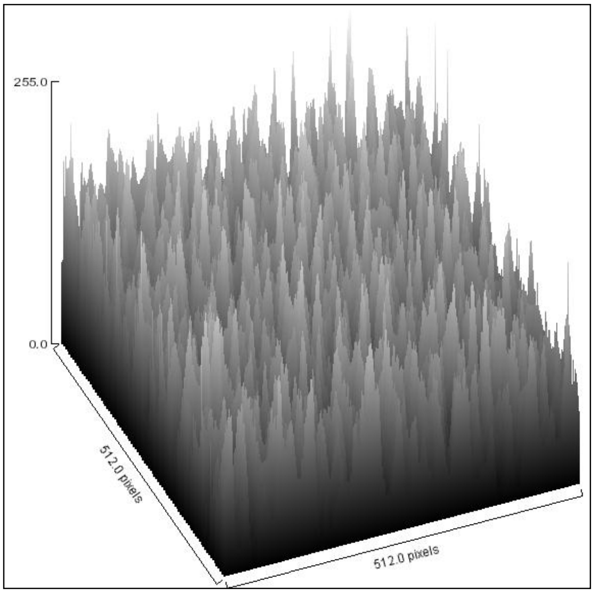

Fig4. Surface plot of the image 248z3.2k

4. Finite element analysis of digital images

On the basis of equivalent images obtained by “ImageJ” (approximation of the actual microstructure observed on SEM micrographs), finite element calculations are performed to estimate the mechanical properties of porous silicon layers. The mesh generation is performed by using the Object Oriented Finite Element Analysis code OOF [9]. The procedure of meshing used in this code generates decreasing elements sizes of the interface regions with different colors, thus allowing separating the contours of the different phases. A two-dimensional model of the mechanical deformation of the specimen is built. An artificial deformation is applied to the structure. Thus, tests of tensile and shear are simulated by applying a series of strains. The results are used to determine the Young's modulus, shear modulus and Poisson's ratio. The behavior law chosen is that of a linear elastic orthotropic material. We used for the calculation 10 equivalent images constructed on 10 SEM micrographs for different areas of the sample to determine the mechanical properties. Five parameters are defined, Young’s modulus E_x and E_y, Poisson’s ratio ν_x and ν_y and shear modulus G. Young's modulus taken in these calculations for silicon is 132GPa and a Poisson's ratio of 0.3. Table1 resume the calculated mechanical properties. Results are in good agreement with those obtained by microindentation. The reduced modulus in microindentation for a maximum load of 300mN ($E_r = E/1\text{-}v^2$) is equal to 52.62GPa ± 6.98% while the result obtained with calculation is 52.82GPa for Young’s modulus, 2.4GPa for shear modulus and 0.21 for the Poisson’s ratio. The results appear to be reasonable.

Table 1. Calculated mechanical properties for PS248 porous silicon

	E_x (GPa)	E_y(GPa)	G(GPa)	v
PS248	38.34	34.43	2.4	0.21

5. Conclusion

An image analysis procedure was developed to transform the micrographs in equivalent images.

The porosity is adjusted and simplified images thus obtained are then used to performing predictive calculation with finite element model.

The finite element described in this paper allowed us to determine the mechanical properties of porous silicon following the linear orthotropic. The results obtained are comparable with experimental measurements.

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Salutation

We would like to salute this research that we dedicate to all those who have an interest in this work.

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